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On the pore size distribution and uniformity improvement in nuclear track filters

Paul Singh*, N Singh and S K Chakarvarti**

Department of Physics, Kurukshetra University, Kurukshetra-132119, India

Abstract : The unique characteristics of nuclear track filters, which entail them more attractive and advantageous than other conventional filters used in a wide variety of applications in the filtration technology, are their easy production, high mechanical stability, high resistance against aggressive chemicals and high doses of radiation etc. besides well defined porosity and high pore-density. In typical applications, the informations on pore size and pore distributions is required which can be obtained by porosimetry tests or SEM techniques. Mean pore size of the filter membranes manufactured by Nuclepore Corporation, USA† are within tolerance of +0% to −20% of rated pore size with ±15% tolerance in pore density. The non-uniformity in tracks may result from the heterogeneity of the material and the statistical fluctuations of the energy-loss rate of the incident ions of well defined energy and isotopic mass and charge. It, therefore, suggests that in order to narrow down the pore size distribution in a plastic nuclear track filter, heterogeneity of the material should be minimized.

In this work we report the effect of thermal annealing prior to etching and pre-etch UV irradiation on the pore size distribution in nuclear track filters of Makrofol N (60 μm, Bayer AG, FRG) irradiated with 15.36 MeV/n normally incident ^{58}Ni ions with a fluence of $\sim 10^{5}/\text{cm}^{2}$ at the UNILAC, Darmstadt, FRG. It is found that in the untreated samples, the etched pore density (etching conditions 6N NaOH, 65°C, 2.5 h) peaks at $0.22 \times 10^{5}/\text{cm}^{2}$ and pore diameter 4.9 to 6.1 with peak at 5.4 μm. After thermal annealing at 90°C for 2 h, the distribution squeezes to 5.9 to 6.7 with peak at 6.4 μm. In case of UV exposure 4.5 h λ=3663 Å) there is a little change in distribution. The effect of liquid surfactant added to the main etchant is also reported on the variation in pore size distribution.

Keywords : Uniformity, tolerance, in-homogeneity, diameter distribution, nuclear track filters.

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1. Introduction

In some typical applications of nuclear track filters (NTFs), the information on the pore size and pore distribution is required. These informations can be obtained by

*Department of Chemistry and Biochemistry, Haryana Agricultural University, Hisar-125004, Haryana India.

**Department of Applied Physics, Regional Engineering College, Kurukshetra-132119, India.

†Nuclepore Corporation, Pleasanton, CA 4566-3294, USA.

porosimetry test or SEM techniques. Nuclear track filter membranes manufactured by (Nuclepore Corporation, USA) have the pore size tolerance of +0% to −20% of the rated pore size with ± 15% tolerance in pore density. This non-uniformity in the pore size may be assigned to various possible reasons :

(a) Heterogeneity of the detector material
(b) Statistical fluctuations of the energy loss rate (Apel 1982)
(c) Inhomogeneity in beam energy

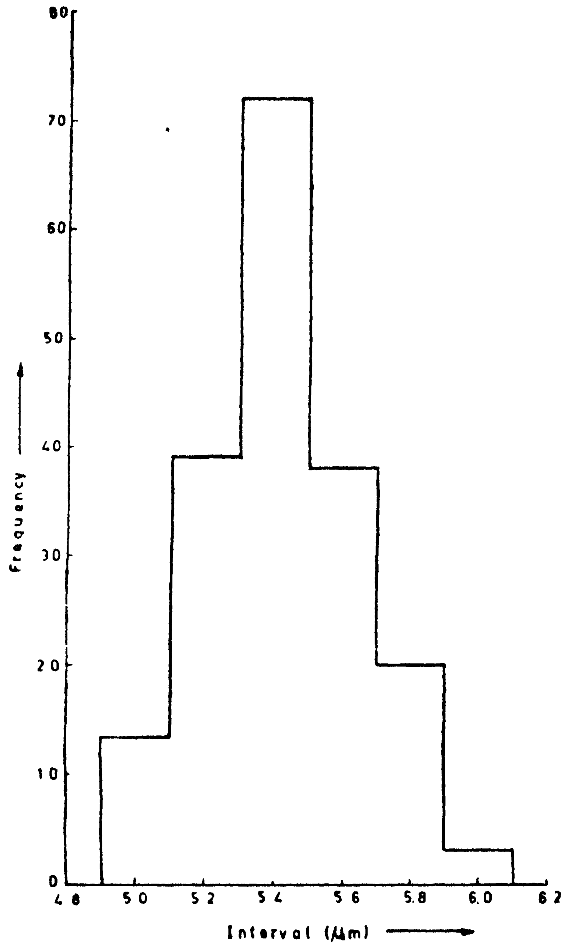

Figure I(a). Histogram of pore diameter distribution in the untreated exposed sample irradiated with normally incident ^{58}Ni

(d) Stochastic nature of irradiation : Multiple holes of varying shape and dispersion in area 2-15% (Riedel and Spohr 1980, Guillot and Rondelez 1981)
(e) Effect of pre-etch treatments such as UV rays, γ-rays and thermal treatment.

It was observed (Guillot and Rondelez 1981) that in case of Makrofol KG NTFs, the two surfaces were not identical and there was a wide difference in the pore size when measured on the two surfaces (dull and shiny). On the UV exposure treatment the pore size distribution is broadened from 10% to 20% for the pores of size 2500 Å after 4 h. exposure (Guillot and Rondelez 1981).

In the present work, we report the effect of pre-etching thermal treatment, UV irradiation and addition of surfactants to the etchant on the pore size distribution in NTFs of Makrofol N (60 μm, Bayer AG, FRG).

2. Experimental

Circular discs (die-cut, dia 5 cm, thickness 60 μm) of Makrofol (from Bayer AG, Lever Kusen, FRG) were irradiated with normally incidence ^{58}Ni ions (15.36 MeV/n) at the Universal Linear Accelerator of Gesellschaft fur Schwerionenforschung at

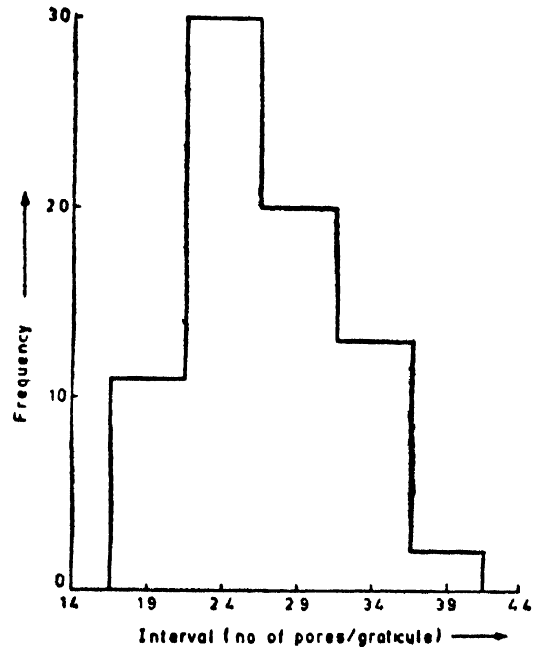

Figure 1(b). Histogram of pore diameter distribution with random particle incidence.

Darmstadt, FRG, with a fluence of $10^5/cm^2$. After about ten months storage under normal laboratory conditions (with no exposure to fluorescent tube lights), the samples were subjected to UV radiation (3663 Å) on both sides for equal intervals, from a UV lamp (WOIAN, 240 V/300 W) at a distance of 15 cm in air from the sample holder whose temperature was maintained constant at 30°C using forced air cooling. Other samples were thermally annealed in an oven for 2 h. at 90°C. To study the effect of surfactants, 0.2% (Vol.) of Ezee (Trade name : Godrej Soap, Bombay) was added to the etchant. All the samples were etched in 6N NaOH at 65°C for 2.5 h, except the case of surfactants, where 6N NaOH+0.2% (Ezee) at 65°C was used. The pore diameters were measured using an optical microscope at 600X and with a least count of ± 0.215 μm.

3. Results and discussion

Figure 1a shows the pore diameter distribution in the untreated but exposed sample etched in 6N NaOH at 65°C. The peak occurs at average value of 5.4 μm. Diameter

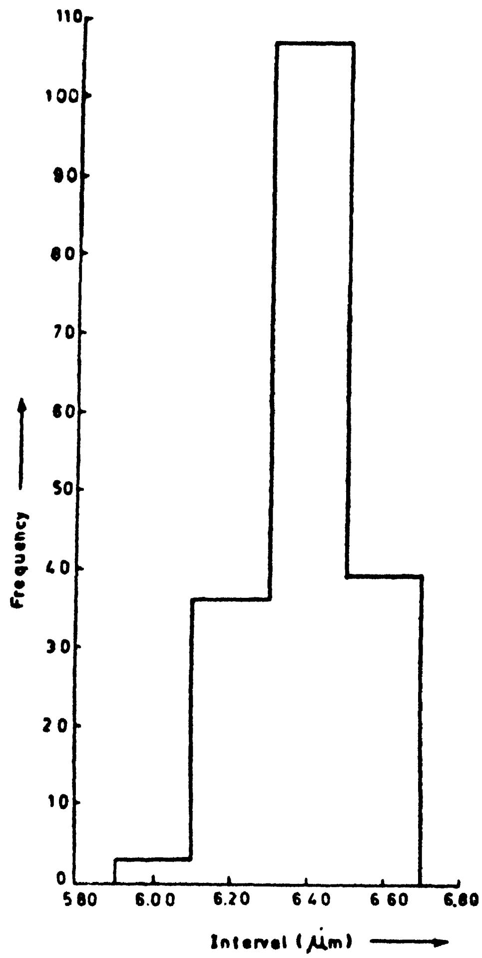

Figure 2. Histogram of pore size distribution for thermally annealed sample.

ranges between 4.9 and 6.1 μm with dispersion of -9.3 to $+13$%. The numbers corresponding to peak form 39% of the total events scanned. Figure 1b shows pore density (measured at 100X, number/frame, frame size=7.5 μm $\times$ 7.5 μm) distribution, giving peak at a value of 24. This is because of randomness in particle incidence.

The pore size distribution for thermally annealed samples is shown in Figure 2. The peak value is now shifted to 6.4 μm (49.8% of total events counted) with variation between the extreme values of 5.9 to 6.7 μm and overall dispersion of -7.8 to $+4.7$%. This clearly suggests the utility of thermal annealing in narrowing down the dispersion in this type of material. Therefore, this demonstrates that thermal annealing might have brought in 'homogeneity' of the material and thus lessened the dispersion.

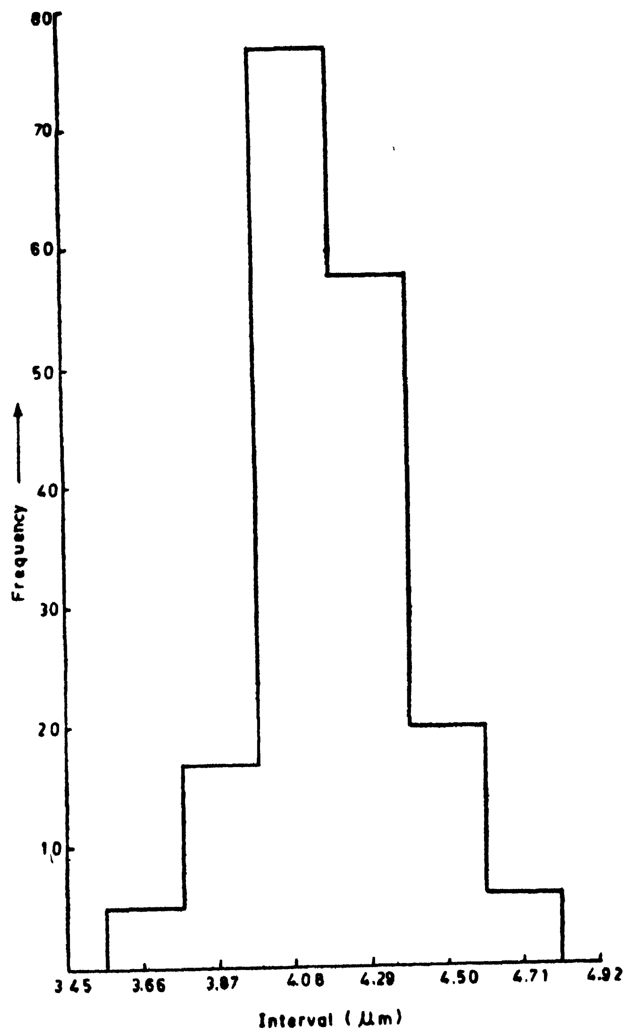

Figure 3. Histogram of pore size distribution for UV irradiated samples.

Figure 3 shows distribution pattern of diameter for UV irradiated samples, peak occurs at 4.1 μm with extreme values between 3.5 and 4.8 μm. 42% of total events scanned falls under the peak and the distribution shows an overall dispersion between -13.2 to 17.6%. Thus UV irradiation distribution is broadened. The effect of surfactant added to etchant does not show any appreciable change in distribution (figure not shown). In this case, peak occurs at 6.02 μm with distribution width of -12.5 to $+12.6$% (38.5% of total events counted fall under the peak).

Concluding from the above studies, it is indicated that a suitable pre-etch thermal treatment of NTFs may prove a technique for optimization of obtaining NTFs with narrow distribution width of etched pores.

References

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